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PETROGRAPHIC METHODS AND THEIR APPLICATION TO THE EXAMINATION OF NONMETALLIC MATERIALS

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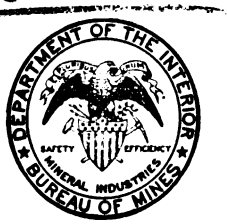

BY

GEORGE T. FAUST AND ALTON GABRIEL

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PETROGRAPHIC METHODS AND THEIR APPLICATION TO THE EXAMINATION OF NONMETALLIC MATERIALS[1]

By George T. Faust[2] and Alton Gabriel[3]

INTRODUCTION

The petrographic microscope plays an important role in the mineral dressing of nonmetallic materials. Petrographers and mineral technologists have evinced an interest in the technique developed, specifically, for mineral-dressing problems. The following dissertation makes the information more generally available.

ACKNOWLEDGMENTS

The authors are indebted to O. C. Ralston, chief engineer, Nonmetals Division, and to Will H. Coghill, supervising engineer of the Southern Experiment Station for a critical reading of the manuscript. They are also indebted to their colleagues for technical information obtained in the course of cooperative work, in particular to J. B. Clemmer for the use of the data on fluorite.

I. ECONOMIC PETROGRAPHY[4]

Microscopic-petrographic methods have been applied successfully for many years in mineral dressing of metallic ores. The work of Schneiderhöhn

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1/ The work upon which this report was based was done under a cooperative agreement between the Bureau of Mines, U. S. Department of the Interior, and the University of Alabama. The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7129."

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4/ "This term is suggested for describing the broad scope of application of microscopic-petrographic methods to mineral products in the following fields: (1) Mineral-dressing products; (2) examination of ceramic materials, including cements; (3) comparison of constituents of pharmaceuticals, industrial chemicals, mine and industrial dusts, and (4) selection of materials for engineering and mining. The term 'economic petrography' thus stands in the same relation to petrography as does economic geology to geology." (See Faust, G. T., A Polarizing Comparison-Microscope for use in Petrographic Measurements: Bureau of Mines Rept. of Investigations 3503, April 1940, 7 pp.

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and Randohr stands out prominently in this field. Full bibliographies of their work and of the work of others are given by Schwartz[5] and Thomson.[6] An interesting application to iron ores is given by Coghill, Howes, and Cooke.[7]

The development of petrography in the last 50 years has yielded not only a mass of descriptive information regarding the constitution of the earth's crust and its probable origin, but it has made also available varied and diverse techniques for the identification and measurement of mineral particles in the composite rocks. The work in sedimentary petrography in particular has been responsible for the development of the petrographic measurements considered in this paper. The works of Krumbein and Pettijohn,[8] Holmes,[9] and of Cayeux[10] are excellent sources for reference to classical methods.

The present paper outlines the methods employed in the petrographic laboratories of the Bureau of Mines Nonmetals Division, and includes pertinent references to the scattered literature on petrographic methods for nonmetallic materials.

The petrographic technique introduced here is by no means new, but rather consists of standard methods that are applied specifically to the solving of problems arising in the assessment and evaluation of nonmetallic materials.

Results of petrographic analyses are used by mineral technologists in planning the method of beneficiation. For purposes of illustration, let us consider two sands, each containing about 1.5 percent ferric oxide. For sand "A" the petrographer reports the presence of the iron-bearing minerals amphibole, pyrite, biotite, and magnetite as the contaminating substances. For sand "B" he reports that all the iron is present as heavy coatings of iron oxide on the surface of the quartz grains. On the basis of these data, it is apparent that the treatment of the two sands will be radically different. Sand "A" would be tabled or magnetically separated to remove the heavy minerals, whereas sand "B" may be discarded or processed by scuff grinding[11] and then subjected to chemical treatment.

5/ Schwartz, G. M., Review of the Application of Microscopic Study to Metallurgical Problems: Econ. Geol., vol. 33, 1938, pp. 440-453.

6/ Thomson, E., History of the Study of Ore Minerals: Am. Mineral, vol. 24, No. 3, 1939, pp. 137-154.

7/ Coghill, W. H., Howes, W., and Cooke, S.R.B., Mineragraphic Aid in the Concentration of Manganiferous Iron Ores: Eng. Min. Jour., vol. 131, No. 8, 1931, pp. 361-365.

8/ Krumbein, W. C., and Pettijohn, F. J., Manual of Sedimentary Petrography: D. Appleton-Century Co., New York, 1938, XIV +549 pp.

9/ Holmes, A., Petrographic Methods: Thomas Murby & Co., London, 1921, 515 pp.

10/ Cayeux, L., Introduction à L'Etude Pétrographique des Roches Sédimentaires: Imprimerie Nationale, Ministerie des Trevaux, Paris, 1916, 524 pp.

11/ DeVaney, F. D., Gandrud, B. W., and Coghill, W. H., Gravity Concentration of Alabama Oölitic Iron Ores: Bureau of Mines Rept. of Invest. 2937, 1939, 7 pp.

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As illustrated later, the petrographic microscope is used extensively to follow the trends in flotation.

II. MICROSCOPIC EXAMINATIONS

Determination of the Principal and Accessory Minerals

Specimens of minerals and rocks occurring in masses large enough to warrant beneficiation studies are first examined to confirm the identity of the supposed valuable minerals and to ascertain their mineralogical associates. To plan the methods of mineral dressing, it is extremely important to know the character of the minerals that are to be rejected in the beneficiation process.

After such a preliminary examination is completed, the mineral technologists submit crushed and screen-sized samples to the petrographer for more complete study.

Since the petrographer is dealing essentially with crushed grains, the powder method, in which temporary mounts are used, has been adopted. This method permits the use of any number of immersion liquids, so that refractive index measurements can readily be made. Furthermore, in temporary mounts grains may be rolled to give desired orientations.

The sized particles usually are 20-mesh (0.833 mm.) down to 200 mesh (0.074 mm.) and, in addition, include the -200-mesh fraction. At this point, before microscopic study, there is some variation in technique. The sized samples either are examined as received or are first subjected to sink-and-float separations on heavy liquids of suitable specific gravity, of which acetylene tetrabromide and methylene iodide are standard members. Each sized fraction is next examined to determine the extent to which the desired mineral is locked (attached) to gangue minerals. Usually, the extent of locking decreases as the particle size decreases. Thus, table 1 indicates the result of such a study on a typical kyanite rock.

TABLE 1. - Mesh of liberation of a kyanite rock

Mesh		
+ 48		Kyanite heavily locked to mica and quartz.
- 48	+ 65	About 30 percent of the kyanite locked to mica and quartz.
- 65	+100	About 8 percent of the kyanite locked to mica and quartz.
-100	+150	Less than 1 percent of the kyanite locked to mica and quartz.
-150	+200	Locking is negligible.
-200		Do.

It is evident that crushing through 100 mesh is sufficient to liberate the kyanite. This screen size is commonly referred to as the "mesh of liberation." Crushing this material below 100 mesh is unnecessary.

Finding the mesh of liberation is one of the most important petrographic studied, for it determines whether or not beneficiation of a given rock

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is possible. Further, it indicates the type of mineral dressing that will be suitable. This is made more apparent by a study of table 2, in which are given the approximate size ranges that can be treated by the respective beneficiation methods.

TABLE 2. - General size limits for mineral-dressing methods

Agglomerate tabling....	20 to 48 mesh	(0.833-0.295 mm.)
Tabling.....	8 to 200 mesh	(2.362-0.074 mm.)
Flotation.....	48 to 325 mesh	(0.295-0.043 mm.)
Jigging.....	4-inch down to 20 mesh	(10.16 cm. -0.833 mm.)

A type of locking that in general precludes beneficiation is the presence of fine inclusions. The inclusions may be ferriferous and as such would introduce the unwanted element, iron. Thus, kyanite crystals that are "peppered" with hematite, magnetite, or ilmenite are useless for ceramic purposes. The discovery of these impurities eliminates the possibility of beneficiation. A kyanite-sillimanite rock from Taos, N. Mex., was rejected because the kyanite grains were peppered with minute grains of magnetite. Quartz with grains containing rutile was similarly rendered useless for certain fields. Theoretically, at least, it is possible to crush or grind a sample so fine that the very small inclusions are liberated. However, the size of the greater portion of the particles might be so reduced in grinding as to make the resultant "feed" not amenable to froth flotation or any other type of concentration. In other words, there is an economic limit to grinding.

Locking is readily detected under the microscope, for it can be observed whether or not the grains are attached to one another. If there is any doubt, it is merely necessary to tap the cover glass with a pointed instrument. This causes the particles to move so that their common or independent action may be noted.

There are certain rocks that, when crushed, yield minerals not easily recognized by the usual microscopic methods. This may be due to a similarity in optical properties, color, or structural features, such as very fine-grained aggregates. Thus, rapid identification of mixtures of quartz and a plagioclase of the oligoclase-andesine type is hindered by the closeness in refractive indexes of the two constituents. This problem has been treated successfully by staining methods.[12]

Likewise, staining methods can be used in evaluating the extent of locking of clayey constituents in phosphate rock, marls, and similar types of fine-grained aggregates.[13] The recent book by Gaudin[14] describes a

12/ Gabriel, A., and Cox, E. P., A Staining Method for the Quantitative Determination of Certain Rock Minerals: Am. Mineral. vol. 14, No. 8, August 1929, pp. 290-292.

13/ Faust, G. T., Staining of Clay Minerals as a Rapid Means of Identification in Natural and Beneficiated Products - With a General Discussion on Staining Technique; Bureau of Mines Report of Investigations 3522, 1940, pp.

14/ Gaudin, A. M., Principles of Mineral Dressing: McGraw-Hill Book Co., Inc., New York, 1939, 554 pp.

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concise treatment of locking as it applies to mineral dressing. An elementary manual describing the ore-testing methods used by the Bureau of Mines has been published by Dietrich and associates.[15]

Structural Features Influencing Grinding and Crushing

Some crushed rocks yield unusual characteristics. Thus, a sillimanite-biotite-quartz rock when ground in a set of rolls produced a ribboning effect. When these ribbons were examined, they were found to consist of biotite flakes firmly pressed together.

The presence of undulatory extinction (indicating a strained state) in quartz and other minerals should be assessed by the mineral technologists, for such materials might be ground easier, owing to the strained condition.

In the crushing of carbonate-bearing rocks, the carbonates, because of their excellent and ready rhombohedral cleavage, tend to concentrate in the fines, and thus appear with the slimes in wet beneficiation processes.

III. SPECIAL PETROGRAPHIC METHODS

The Use of Heavy Liquids in Specific-Gravity Separations

Carbon tetrachloride with a specific gravity of 1.595 is used for the partial separation of the noncarbonaceous materials from coal in coal products. It is also used in separating what is commonly termed "oilrock" from fluorite in the Illinois-Kentucky fluorite rocks.

Acetylene tetrabromide (specific gravity 2.96) is the most useful liquid for many nonmetallic separations. For concentration of the heavier constituents, separations on methylene iodide (specific gravity, 3.32) are satisfactory. If further separation beyond 3.3 gravity is required, it is necessary to resort to the use of thallium salts, and the sink-and-float separations are made according to the standard methods described by Sullivan,[16] by O'Meara and Clemmer,[17] and by Vassar.[18] Not only do such separations affect the concentration of the heavier constituents but also afford density data useful in mineral identification.

15/ Dietrich, W. F., Engel, A. L., and Guggenheim, M., Ore-dressing Tests and Their Significance: Bureau of Mines Rept. of Investigations 3328, 1937, pp. 5-35.

16/ Sullivan, John D., Heavy Liquids for Mineralogical Analyses; Bureau of Mines Tech. Paper 381, 1927, 26 pp.

17/ O'Meara, R. G., and Clemmer, J. B., Methods of Preparing and Cleaning Some Common Heavy Liquids Used in Ore Testing: Bureau of Mines Rept. of Investigations 2897, 1928, 6 pp.

18/ Vassar, Helen E., Clerici Solution for Mineral Separation by Gravity: Am. Mineral., vol. 10, 1925, pp. 123-125.

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For microdensity determination, the recent technique of Jahns,[19] employing Clerici solution, is readily applicable.

Magnetic Separations and Observations

The Frantz isodynamic separator is capable of very sensitive magnetic separations. Thus, it is possible to separate minerals carrying iron-bearing inclusions from those free of such inclusions. It is also employed to separate iron-bearing impurities in sands and crushed grains, especially where the impurities occur only as occasional grains and where concentration is advantageous for microscopic identification or chemical or spectrographic analysis. Muscovite can be separated from iron-free feldspar and quartz.

Under the microscope, strongly magnetic substances, particularly magnetite, are detected by passing a magnetized needle over, but not touching, the surface of the preparation while observing it through a 16X objective. Magnetite and metallic iron are moved by the needle and may form chains with other such magnetic grains.

Electrostatic separations

Nonmetallic mineral grains may be separated from feebly magnetic impurities by virtue of a difference in conductivity. Thus, pyrite, which is only weakly magnetic, is readily removed by electrostatic means. Kyanite concentrates may contain small quantities of pyrite, which may be removed by several repeated electrostatic separations.

The Crook electrostatic[20] separator, consisting of two copper plates connected to a Wimshurst influence machine, has been used with success in separating kyanite from pyrite, quartz from pyrite, and other similar mixtures.

Centrifugalizing Small Amounts of Materials in Heavy Liquids

The microcentrifuge is used when normal heavy liquids do not give good separations in a short time. This usually occurs when the concentration of the impurities is small in the original sample or when the specific gravity does not differ widely.

In searching for heavier constituents in coal, such as pyrite, marcasite, and siderite, a satisfactory separation may be obtained by first making a preliminary separation on carbon tetrachloride and then centrifugalizing in a microcentrifuge with acetylene tetrabromide. Rutile, zircon, staurolite, kyanite, and other heavy minerals occurring sparsely in clays may

19/ Jahns, R. H., Clerici Solution for the Specific-gravity Determination of Small Mineral Grains: Am. Mineral, vol. 24, 1939, pp. 116-122.

20/ Crook, Thos., Economic Mineralogy: Longmans Green & Co., London, 1921, pp. 108-110.

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also be roughly concentrated first by decantation or other gravity methods, and then settled in the microcentrifuge. The "float" in the centrifuge tube is removed by adding more liquid, which produces an overflow, carrying the float with it down the outside of the tube into a small beaker.

Sampling With an Electric Drill

Small inclusions or other small areas in rocks or polished sections of rocks can be removed for microscopic or spectrographic examination by drilling out the inclusion as powder with a dental drill under a binocular microscope. One type of drill in use consists of a hand piece (which carries the drills) attached to a flexible cable about 2 feet long. The drill is driven by a 1/20-horsepower motor at about 500 r.p.m. Dental drills of steel can be obtained in virtually all shapes and sizes to at least 0.5 mm. diameter. For hard material (+5 in Mohs' scale of hardness), drills of carboloy or diamond point are used. These drills can be obtained from Arthur A. Crafts Co., of Boston, Mass.

Microchemical and Spectrographic Tests

When a spectrograph is available, it is used exclusively to identify the cations, since it is more rapid than microchemical tests. A trained spectroscopist can estimate fairly accurately the cationic elements present. A spectral analysis for the principal elements gives the composition of the material - a help in identifying those materials not easily identified petrographically.[21] For example, fibrous talc, sericite, and pyrophyllite of small particle size often are difficult to identify specifically by petrographic means. If by spectrographic analysis magnesium is found to be present and alumina low or absent, fibrous talc is the only constituent; if the magnesium is absent and potash is present, sericite is a constituent; and if both potash and magnesium are absent, the material is pyrophyllite. It is difficult to identify mixture of serricite and pyrophyllite by other than quantitative analysis or possibly X-rays.

Chlorine, bromine, iodine, the rare gases, sulfur, selenium, tellurium, oxygen, and nitrogen cannot be identified with an ordinary spectrograph. If their presence is suspected, they as well as other anions, are detected by microchemical tests.

Solubility Separations

Constituents may be present in rocks in such small quantities as to escape detection or be masked by the more predominant minerals. If the rocks are acid-soluble, such as limestones, dolomites, and marls, the sample may be treated with a weak acid, such as citric or acetic, and the

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21/ Lee, O. I., and Wright, Thos. A., On Identifying Minerals With the Aid of the Spectrograph: Proc. 6th Summer Conf. on Spectroscopy and Its Application, John Wiley & Sons, Inc., New York, 1939, pp. 38-46.

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residue separated for examination. An Alabama marl that consisted of intimately locked calcite and clay was easily cleaned with acids and the clay content found to be kaolinitic in type and to constitute 10 percent of the sample.

Phosphate rock, coal, salt, and other materials likewise may be cleaned by means of suitable solvents. More chemically resistant materials require more drastic treatments. Thus, a mixture of quartz, biotite, and sillimanite was treated first with sulfuric acid and then with hydrofluoric acid, which left a residue of sillimanite.

Iron oxide coating usually is removed by simple digestion with hydrochloric acid. If the use of strong acids is not feasible and if the particles are small enough in size to be attacked actively, the method of Drosdoff and Truog[22] may be used. This consists of treating a suspension of the coated minerals with hydrogen sulfide, followed by solution of the iron sulfides in 0.05 N hydrochloric acid.

Refractive-Index Media

The well-known liquid refractive-index mediums, ranging from water with a refractive index of 1.333 to a methylene iodide-sulfur mixture with a refractive index of 1.780, are used. Possession of either Merwin's solutions[23] or the newly proposed phosphorus liquids of West[24] also is desirable. For minerals of high refractive index it is necessary to resort to the mixtures of arsenic and antimony iodides in piperine and sulfur-selenium melts.

Staining Methods

The use of stains for mineral differentiation in quantitative estimation of the components in rocks is well established in petrographic technique. For a general discussion of staining technique the reader is referred to a forthcoming paper by Faust.[25]

The technique of Gabriel and Cox[26] is employed for estimating quartz-feldspar mixtures. It requires exposure of the grains to hydrofluoric acid

22/ Drosdoff, M., and Truog, E., A Method for Removing Iron Oxide Coating from Minerals: Am. Mineral., vol. 20, No. 9, September 1935, pp. 669-673.

23/ Merwin, H. E., Media of High Refraction: Jour. Washington Acad. Sci., vol. 3, 1913, pp. 35-40.

24/ West, C. D., Immersion Liquids of High Refractive Index: Am. Mineral., vol. 21, No. 4, April 1936, pp. 245-250.

25/ Faust, G. T., The Staining of Clay Minerals as a Rapid Means of Identification in Natural and Beneficiated Products - with a General Discussion on Staining Technique: Bureau of Mines Rept. of Investigations 3522, 1940, 21 pp.

26/ Gabriel, A., and Cox, E. P., A Staining Method for the Quantitative Determination of Certain Rock Minerals: Am. Mineral., vol. 14, No. 8, August 1929, pp. 290-292.

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vapors, followed by staining in a concentrated sodium cobaltinitrite solution. The potash feldspars are stained yellow, the plagioclases are rendered opaque to transmitted light, whereas the quartz remains uncolored and clear. Untwinned oligoclase-andesine has properties similar to those of quartz, and staining methods offer the easiest means of differentiation.

The technique of Shand[27] may be used to distinguish feldspathoids. Keith[28] employs selective staining which involves a combination of staining methods before Rosiwal analysis. Head and Crawford[29] use potassium dichromate to identify cerusite and anglesite in products from mineral dressing. For the detection of clay in diatomaceous earth, Merwin[30] recommends malachite green in glycerin. Faust[31] has studied the staining relations of kaolinites and bentonites.

Particle-Size Measurements

Particle size is determined according to standard methods, such as those described by Chamot and Mason.[32] Papers by Roller[33] and Heywood[34] furnish modern viewpoints for interpreting the data. The measurement and interpretation of fine particles are also described by Goldman and DallaValle.[35] A technique devised by Faust and Cooke[36] for measuring the projected area of opaque particles has been used on coal. By the use of suitable filters or by staining, its use may be extended to some nonmetals. The technique serves equally well for calibrating screens.

27/ Shand, S. J., On the Staining of Feldspathoids and on Zonal Structure in Nepheline: Am. Mineral., vol. 24, No. 8, August 1939, pp. 508-513.
28/ Keith, M. L., Selective Staining to Facilitate Rosiwal Analysis: Am. Mineral., vol. 24, No. 9, September 1939, pp. 561-565.
29/ Head, R. E., and Crawford, A. L., A Staining Method for Distinguishing Cerusite and Anglesite in Ores, Concentrates, and Tailings: Bureau of Mines Rept. of Investigations 2932, 1929, 3 pp.
30/ Calvert, Robert, Diatomaceous Earth: Chemical Catalog Co., New York, 1930, p. 71. (Letter to Calvert from Merwin.)
31/ Faust, G. T., Work cited in footnote 26, p. 14.
32/ Chamot, E. M., and Mason, C. W., Handbook of Chemical Microscopy: John Wiley & Sons, Inc., New York, 1938, chap. 13, pp. 408-431.
33/ Roller, P. S., A Classification of Methods of Mechanical Analysis of Particulate Materials: Proc. Am. Soc. Test. Mat., vol. 37, pt. 2, 1937, pp. 675-683.
34/ Heywood, H., Measurement of the Fineness of Powdered Materials: Inst. Mech. Eng., 1938, pp. 1-52.
35/ Goldman, F. H., and DallaValle, J. M., An Accurate Method for the Determination of the Components of a Heterogeneous Particulate Mineral System: Am. Mineral., vol. 24, 1939, pp. 40-47.
36/ Faust, G. T., and Cooke, S.R.B., Ocular Photocell for the Rapid Determination of Projected Area of Opaque Particles: Bureau of Mines Rept. of Investigations 3460, 1939, 14 pp.

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A word should be added regarding screens, as they offer a rapid and convenient means of sizing material for microscopic examination. Screens of 3-inch diameter are suitable, and the have the additional advantage of greater uniformity in mesh than corresponding sieves of larger diameter. It is not well-known that screens of 400-mesh to the linear inch are available. For accurate sizing, analysis sieves should be chosen upon the basis of microscopic examination. The present practice for fine materials is first to sieve the sample wet on 400 mesh, dry, and resieve the oversize on appropriate screens.

The Haultain Superpanner

This instrument was devised by Haultain[37] for concentrating very fine telluride particles existing in exceedingly small proportions in the ore. It may also be used for concentrating rarer constituents. Samples of minerals such as monazite, which are present in very small quantities, have been readily concentrated in amounts large enough to be identified microscopically.

IV. EXAMINATION OF BENEFICIATED MATERIALS

Cleaning Treated Minerals

Products resulting from such mineral-dressing operations as jigging, tabling, screening, classification, and magnetic separation require no special treatment before microscopic examination. However, the products resulting from flotation, agglomerate tabling, and heavy-liquid separations, on the other hand, usually require some treatment to remove films of organic compounds, which may otherwise interfere with identification or alter the properties of the immersion liquids.

Many flotation reagents are soluble in water, hence they are easily removed by washing. Those not soluble in water usually may be removed by treating them with common solvents, such as alcohol, acetone, benzene, kerosene, carbon tetrachloride, ether, or petroleum ether.

Sulfides that have been floated by "Aerofloat" reagent (P_2S_5 in cresylic acid) may be difficult to identify because of the surface coating. This coating may be destroyed by shaking the grains in a dilute solution of hydrofluoric acid.[38]

Wetting solutions, such as saponin or soap, have been suggested by Coghill and Bonardi[39] for use in handling flotation concentrates badly "gummed with oil" by allowing them to be sized by screening before microscopic

37/ Haultain, H. E. T., Splitting the Minus-200 Mesh with the Superpanner and the Infrasizer: Canadian Min. and Met. Bull., vol. 40, May 1937, pp. 229-240.

38/ Suggested by our colleague, B. H. Clemmons, of the Southern Experiment Station, Bureau of Mines.

39/ Coghill, W. H., and Bonardi, J. P., Approximate Quantitative Microscopy of Pulverized Ores: Bureau of Mines Tech. Paper 211, 1919, pp. 6-7.

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study. Graphite flotation concentrates may be cleaned[40] by standing in ether for about 30 minutes. Flotation froths are examined, according to Gaudin[41] by "vanning a small portion of a froth in a watch glass with acetone as the vanning liquid. The practice is superior to that of vanning in water, as the viscosity of acetone is much lower and as the froth breaks down readily in excess acetone."

The washed products are dried over a hot plate. Minerals that undergo dehydration, dissociation, or polymorphic transformation at relatively low temperatures should be heated with care. Gypsum concentrates undergo dehydration when heated to too high a temperature.

Methods of Estimation

The percentage composition of a heterogeneous mixture of particles may be estimated either by visual inspection with ocular nets or by mechanical devices. Emery and De Chicchis[42] have surveyed the earlier methods up to 1925.

Visual estimation is based largely upon experience. The longer a petrographer works with a given type of ore, the more proficient he becomes in estimating its constituents. In general, the highest accuracy is obtained on rocks whose minerals are similar in size, shape, and hardness. Minerals that occur in heterogeneous mixtures as thin plates, such as the micas and chlorites, and acicular or lath-shaped crystals, such as sillimanite are likely to be overestimated. Sillimanite, in particular, may occur as a fasciculate bundle of fibers entirely enclosing a quartz core. A primary requirement for visual estimation is that the grains be sized closely.

Isotropic minerals such as fluorite or garnet may be estimated more accurately by the use of a rotating analyzer or polarizer. The analyzer usually is rotated from 5 to 15°. This permits both isotropic and anisotropic grains to be seen at the same time. The isotropic grains transmit enough light to outline their borders; the anisotropic grains exhibit interference colors. Insertion of the selenite plate between crossed nicols gives a similar effect.

The accuracy obtainable is illustrated in table 3, which compares the visual microscopic estimates with those based upon chemical analysis for alumina. The ore is a crushed pegmatite from North Carolina made up essentially of microcline, quartz, and a small amount of plagioclase. The microcline content was calculated by converting the percentage of chemically determined alumina directly to microcline.

40/ Dub, G. D., and Moses, F. G., Mining and Preparing Domestic Graphite for Crucible Uses: Bureau of Mines Bull. 112, 1920, p. 45.

41/ Gaudin, A. M., in a letter to E. Leitz, dated Oct. 13, 1932, and quoted in "What Scientists Say of Leitz Ultropak"; New York, Booklet 17, p. 14.

42/ Emery, A. H., and De Chicchis, R., Quantitative Mineralogical Analysis of Rock-Dusting Materials and Survey of Some Coal-Measure Shales of Western Pennsylvania: Carnegie Inst. Technol. Bull. 21, 1925, pp. 8-12.

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In a similar manner, table 4 shows the relationship between the visual microscopic estimate and the analytical calculations for a kyanite rock from California. The product studied was a kyanite concentrate. The agreement in both illustrations is satisfactory.

TABLE 3. - Comparison of visual microscopic estimates with those based upon chemical analysis

Beneficiation product	Visual microscopic estimate	Content based upon alumina determination
Flotation tailing.....	Feldspar = 0.5% ± Quartz = remainder	Feldspar = 0.55% Quartz = remainder
Flotation concentrate..	Quartz = 1 to 2% ± Feldspar = remainder	Quartz = 1% Feldspar = 99%
Agglomerate tabling concentrate.....	Quartz = 3 to 4% ± Feldspar = remainder	Quartz = 4% Feldspar = 96%
Agglomerate tabling tailing.....	Feldspar = 5 to 8% ± Quartz = remainder	Feldspar = 4.1% Quartz = remainder

TABLE 4. - Estimates on kyanite concentrate

Mineral	Visual microscopic estimate, percent	Content based upon chemical analysis, percent
Rutile.....	2 to 3	2.5
Kyanite.....	90 to 95	90.8
Quartz.....	5 to 10	6.7
Mica.....	Very small amount.	...
Iron oxides.....	.....	0.92

Accuracy of visual estimation appears to be highest in the range of 0 to 5 percent and diminishes as the mixtures approach the 50 - 50 range.

Counting methods are especially useful in evaluating the composition of multicomponent mixtures. Schneiderhohn[43] has developed a practical method, which is quoted below:

> One of the mixtures consisted of quartz, galena, and chalcocite. The ore was crushed and ground so that it would pass through a hundred mesh but was caught on a hundred and twenty mesh screen. This gave the particles a uniformity of size. The sample was well mixed, and then a small amount spread out on a glass slide. A counting cell assisted in the counting. The number of quartz, galena, and chalcocite particles appearing was noted and the average of about twenty fields taken. If x, y, and z represented the average number of quartz, galena, and chalcocite particles, respectively, and d, d' and d" their respective densities, the percentage of each present is found from the following formulae.

43/ Lindsley, L. C., Industrial Microscopy: Wm. Byrd Press, Richmond, Va., 1929, p. 239.

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$$SiO_2 \text{ quartz} = \frac{dx}{dx + d'y + d''z} \cdot 100$$

$$PbS \text{ galena} = \frac{d'y}{dx + d'y + d''z} \cdot 100$$

$$Cu_2S \text{ chalcocite} = \frac{d''z}{dx + d'y + d''z} \cdot 100$$

When the percentage of galena, quartz, and chalcocite is known, the percentage of lead and copper can easily be figured. It is interesting to know that in test cases the method checked with the actual analysis very closely, and the time consumed was only a few hours, while the older method required several days.

Mechanical devices, such as the Hunt-Wentworth[44] recording micrometer, the Dollar integrating micrometer,[45] and the Hurlburt electric counter,[46] may be used to ascertain the percentage composition of a heterogeneous mixture. Because of the thickness factor and the fact that the grains will tend to be oriented parallel to their length, it is necessary to work with sized fractions. Recently Goldman and Dalla Valle[47] have proposed a method for assessing mixtures heterogeneous in both mineral content and particle size.

An interesting method for assessing the monazite content of pegmatite samples is described by Schaller.[48]

Comparison microscopes are very helpful in evaluating the purity of successive products resulting from beneficiation studies. They are far superior to the usual practice of forming a mental picture of one slide on the stage of the microscope and then attempting to compare this mental image with the actual image of a second slide. A polarizing comparison-microscope has been developed,[49] which greatly extends the applicability of comparison microscope measurements.

44/ Hunt, W. F., An Improved Wentworth Recording Micrometer: Am. Mineral, vol. 9, 1924, pp. 190-193.

45/ Dollar, A. T. J., An Integrating Micrometer for the Geometrical Analysis of Rocks: Mineral Mag., vol. 24, 1937, pp. 577-594.

46/ Hurlburt, C. S., Jr., An Electric Counter for Thin-Section Analysis: Am. Jour. Sci., vol. 237, April 1939, pp. 253-261.

47/ Goldman, F. H., and Dalla Valle, J. M., An Accurate Method for the Determination of the Components of a Heterogeneous Particulate Mineral System: Am. Mineral., vol. 24, 1939, pp. 40-47.

48/ Schaller, W. T., A Large Monazite Crystal from North Carolina: Am. Mineral., Vol. 18, October 1933, pp. 435-440.

49/ Faust, G. T., A Polarizing Comparison-Microscope for Use in Petrographic Measurements: Bureau of Mines Rept. of Investigations 3503, 1940, 7 pp.

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Petrographic Measurements as an Aid in Following the Trend of Flotation Processes

Table 5 presents the results of detailed microscopic analyses by the method of counting on well-sized flotation products resulting from a study of the efficiency of a flotation process.

TABLE 5. - Detailed microscopic analysis of fluorite in sphalerite concentrates from an Illinois deposit

Sample No.	Screen size	Microscopical results (percent)			Weight percent, fluorite	Analytical data	
		Quartz	Sphalerite	Carbonate		Fluorite, percent	Carbonate, percent
1.....	+200	9.8	77.5	0.8	11.9	11.36	0.82
1a....	-200+400	-	94.9	-	5.1	4.69	.33
2.....	+200	5.1	91.8	0.2	2.9	3.34	.20
2a....	-200+400	.8	98.1	.1(-)	1.0	1.16	.14
3.....	+200	5.4	92.0	.1(-)	2.5	2.29	.17
3a....	-200+400	.5	99.1	Trace	.4	.58	.14
4.....	+200	5.3	92.8	0.1 (-)	1.8	1.84	.15
4a....	-200+400	.9	98.8	Trace	.3	.37	.10

TABLE 6. - Distribution of fluorite in float on liquid of 3.3 specific gravity

Sample No.	Locking relations of the fluorite			Fluorite content of the sample, percent
	Locked	Free	Speckled	
1.....	60.3	0.7	39.0	11.93
1a.....	48.6	.7	50.7	5.14
2.....	79.0	-	21.0	2.92
2a	79.0	0.4	20.6	1.03
3.....	93.7	-	6.3	2.55
3a.....	92.2	-	7.8	.42
4.....	93.5	-	6.5	1.79
4a.....	94.7	-	5.3	.25

A very large number (average 900 to 1,000) of particles was counted in each product. The agreement between the values for fluorite obtained microscopically and analytically is good. The carbonate values agree quite well for the larger quantities but not for smaller quantities. This may be due to the fact that small amounts of very fine calcite escaped detection. Calcite has such a marked and ready cleavage that grinding concentrates it in the finer sizes.

The locking relationships of the fluorite that floats on methylene iodide with a specific gravity of 3.3 are presented in table 6. The "locked fluorite" consists principally of fluorite grains locked to sphalerite and in part to "oil rock" and perhaps a small amount of galena. The "speckled fluorite" consists of fluorite grains that have small specks

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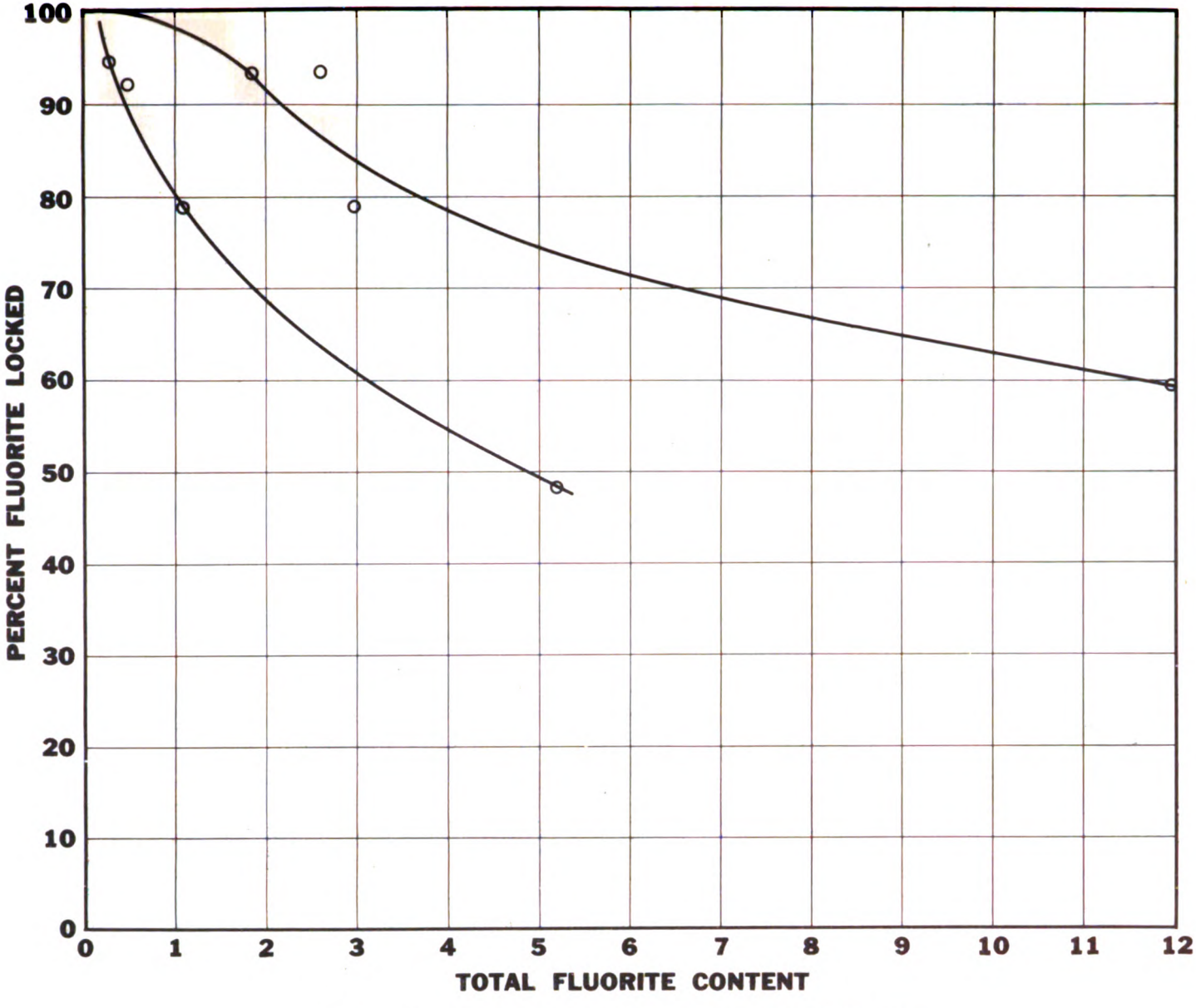

Figure 1.—Ratio of locked fluorite to total fluorite.

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of an opaque substance on their surfaces. This opaque substance is probably oil rock (residues from the evaporation of an oil) in all instances, although it was not possible to prove this by microscopic means.

The quartz is locked principally to oil rock, although occasional locking to sphalerite was noted. From an examination of table 6 it is apparent that the ratio of locked fluorite to free fluorite increases as the total percentage of fluorite in the sample decreases. This is shown in figure 1. Curve A is based upon the data for + 200-mesh material. Curve B represents the results for -200- + 400-mesh particles.

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